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Effects of Atmosphere on the Tribological Properties of a Chromium Carbide Based Coating for Use to 760 °C

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EFFECTS OF ATMOSPHERE ON THE TRIBOLOGICAL PROPERTIES OF A CHROMIUM CARBIDE BASED COATING FOR USE TO 760 °C

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SUMMARY

The effect of atmosphere on the tribological properties of a plasma-sprayed chromium carbide based self-lubricating coating is reported in this paper. The coating contains bonded chromium carbide as the wear resistant "base stock" to which the lubricants silver and barium fluoride/calcium fluoride eutectic are added. It has been denoted as NASA PS200. Potential applications for the PS200 coating are cylinder wall/piston ring couples for Stirling engines and foil bearing journal lubrication. Friction and wear studies were performed in helium, hydrogen, and moist air at temperatures from 25 to 760 °C.

In general, the atmosphere had a significant effect on both the friction and the wear of the coating and counterface material. Specimens tested in hydrogen, a reducing environment, exhibited the best tribological properties. Friction and wear increased in helium and air but are still within acceptable limits for intended applications.

A variety of x-ray analyses was performed on the test specimens in an effort to explain the results. The following conclusions are made:

(1) As the test atmosphere becomes less reducing, the coating experiences a higher concentration level of chromic oxide at the sliding interface which increases both the friction and wear.

(2) Beneficial silver transfer from the parent coating to the counterface material is less effective in air than in helium or hydrogen.

(3) There may be a direct relationship between chromic oxide level present at the sliding interface and the friction coefficient.

E-3311

INTRODUCTION

The lubrication of sliding contacts at high-temperatures in reactive environments is a major challenge in the development of advanced heat engines (e.g., Stirling engines, adiabatic diesel, turbomachinery, etc. (ref. 1)). In an effort to alleviate this problem a plasma-sprayed chromium carbide based coating, PS200, has been developed at NASA Lewis Research Center (refs. 2 to 4). Specific applications include the lubrication of piston ring/cylinder wall couples and foil bearing journals. The base material of this coating system is metal bonded chromium carbide. It is a good candidate for high temperature wear applications because of its thermal and chemical stability and excellent wear resistance. However, when used alone as a coating for sliding contacts it displays poor friction characteristics. By adding the solid lubricants silver and barium fluoride/calcium fluoride eutectic to the chromium carbide base coating its friction and wear properties can be significantly improved (ref. 5). Silver, because of its low shear strength, is a good thin film lubricant at low temperatures and the eutectic, which undergoes brittle to ductile transition around 500 °C, is a good lubricant at high temperature (ref. 6). Thus the coating is designed to lubricate over a wide temperature range from low temperature starts to 900 °C.

Previous tests with this coating system, specifically a coating designated PS200 (80 wt % bonded Cr_3C_2, 10 wt % Ag, 10 wt % BaF_2/CaF_2) indicated that the friction and wear behavior of the coating is significantly influenced by the test atmosphere (ref. 5). Wear data varied approximately an order of magnitude depending on the test atmosphere. Test atmospheres used were hydrogen, helium, and moist air. This paper describes a program to further systematically study the influence of atmosphere on tribological performance and to develop a mechanism to understand this behavior.

EXPERIMENTAL MATERIALS

Test Specimen Configuration

A pin-on-disk type friction and wear tester was used in this study. With this apparatus, a normal load is applied to a clean metal pin that is in sliding contact with a rotating disk coated with PS200.

Wear Pins

The wear pins used in this program are made of a hardened cobalt alloy. It is chosen because of its strength at elevated temperatures, chemical and thermal stability, and suitability for use as an engine component material. This cobalt alloy gave consistent, repeatable friction and wear results when slid against the PS200 coating in a previous study (ref. 5). The test pins are hemispherically tipped with a radius of 0.476 cm and are 2 cm long. The nominal composition of the pins by weight percent is: 59 Co, 30 Cr, 4 W, 2 Ni, 5 other. The Rockwell hardness is 40C. The surface finish of the pins is 6 μin. rms.

Coating

The PS200 coating consists of 80 wt % metal-bonded chromium carbide and 10 wt % each of silver and barium fluoride/calcium fluoride eutectic. The coating components are prepared in powder form then physically mixed together prior to plasma-spray application onto test disks. Table I gives the mesh sizes and composition of the PS200 coating components.

Most of the coating specimens used in this study were prepared by simply mixing the individual powders together prior to plasma spraying. Recently, it was found that both the tribological performance and compositional uniformity of the coating were improved by prefusing and regrinding the eutectic ratio of fluorides prior to mixing with the other powders. Measurable improvements in the friction and wear properties of the coating are achieved using this improved processing step. Any comparison of the data from the earlier experiments to data from coatings made by the improved method should take this point into consideration. Coatings made with the prefused eutectic can be expected to show improved tribological characteristics in all atmospheres.

Test Atmosphere

The test atmosphere is bottled hydrogen, helium, or moist air. Purities of the hydrogen and helium are 99 and 99.997 percent respectively. The moist air is supplied from dried, filtered service air routed through a deionized water filled bubble jar. The relative humidity of the air entering the test chamber is measured with an electronic humidity meter. The relative humidity of the air supplied to the test chamber is 35 percent at 25 °C throughout the tests.

Coating and Finishing Procedure

The test disks are 6.35 cm in diameter, 1.27 cm thick, and made of a high temperature precipitation-hardened nickel chromium alloy hardened to Rockwell 40C. The disk composition by weight percent is: 70 Ni, 15 Cr, 7 Fe, 2.5 Ti, 1 Al, 1 Mn, 1 Co, 2.5 other. The disks are first sandblasted, then a thin bond coat (0.0076 cm) of nichrome (80 wt % Ni, 20 wt % Cr) powder is plasma-sprayed onto the roughened surface. The PS200 powder mixture is plasma-sprayed onto the bond coat to a thickness of about 0.038 cm. The spraying parameters are given in table II. The coating is then diamond ground to give a total coating thickness (bond coat plus lubricant coat) of 0.025 cm. Appendix A describes the grinding procedure. During grinding, particular care must be taken to prevent smearing of the coating and to prevent selective removal of the soft phases. These problems can be avoided by employing the recommended depths of cut with a dressed diamond wheel. Lapping procedures should be especially avoided because of their tendency to preferentially remove the soft lubricant phases (silver and fluoride eutectic).

APPARATUS AND TEST PROCEDURE

A pin-on-disk apparatus is used in this study (fig. 1). A hemispherically tipped pin is loaded against a disk by means of dead weights. The normal load is 0.5 kg. Friction force is continuously measured by means of a temperature compensated strain gauge bridge. The pin generates a 51 mm diameter wear track on the disk. Sliding is unidirectional and the velocity in these experiments is 2.7 m/s. The specimens are heated by a low frequency induction coil. Disk surface temperatures are monitored on the wear track 90 ° ahead of the sliding contact with an infrared pyrometer capable of measuring temperatures from 100 to 1400 °C with 5 percent accuracy.

Prior to each test, the coated disks are heated in a vacuum oven at 150 °C for 3 hr to remove any volatile residue remaining from the grinding operation and subsequent handling. Both the pin and the disk are then cleaned with ethyl alcohol and scrubbed with levigated alumina. Finally, the specimens are rinsed with distilled water and air dried.

The test duration is 1 hr at each of the three temperatures 25, 350, and 760 °C. Rider wear is measured every 30 min by removing the pin and measuring the resulting circular wear scar on the hemispherical surface from which the wear volume is calculated. Locating dowels on the pin holder assures accurate relocation of the pin. Disk wear is measured after each hour by recording a surface profile across the disk wear track, computing the area of removed/displaced coating, and multiplying by the circumference of the wear track to obtain the wear volume.

The test gases are routed through a flow meter at a rate of 0.014 m^3/min. The volume of the test chamber is 0.002 m^3.

For tests in helium or hydrogen the test chamber is purged for 10 min with nitrogen before the test gas is introduced. Testing is begun after the test gas has purged for 10 min. For tests in air the chamber is closed and the test air is allowed to purge the chamber for 10 min prior to testing the specimens. After elevated temperature tests in helium or hydrogen, the specimens are cooled below 150 °C before opening the chamber to inhibit specimen oxidation which might invalidate later analysis.

Analysis Procedure

One wear disk from each test atmosphere, helium, hydrogen, and moist air, is selected for X-ray Photoelectron (XPS) analysis. A 120° sector is cut from each disk. The three 120° sectors are reassembled to form one "composite" disk (fig. 2). The "composite" disk contains a portion of three different test specimens, each run in different atmospheres. The "composite" disk is placed into the XPS chamber for analysis. By adopting this procedure, coating analysis of three test specimens can be completed without opening the analysis chamber. This practice eliminates errors associated with switching specimens and recalibrating the XPS probe.

Additional samples are prepared from the remaining portion of each disk for elemental analyses by Energy Dispersive Spectroscopy (EDS), and for the identification of crystalline compounds by x-ray diffraction techniques.

EXPERIMENTAL RESULTS

The results of the friction and wear experiments are summarized in table III and shown graphically in figs. 3 to 5. The data support previous results which indicated that the test atmosphere does significantly affect the friction and wear behavior of the coating. Tests in hydrogen give the lowest friction and wear results. Friction coefficients are generally 0.23±0.05 and coating wear factors (k) are low 6.4×10^{-10} cm^3/(cm-kg). See appendix B for an explanation of the wear factor k. In helium the friction coefficients are 25 percent higher, that is, 0.29±0.03, and the coating wear factor increases by 40 percent. When the coating is tested in air the friction coefficients are slightly higher than in helium, 0.32±0.05 but the coating wear factor increases by 50 percent. Pin wear follows the same trend as the coating wear. In general then, the friction and wear of the coating and counterface material increase as the test atmosphere becomes less reducing in the following order: hydrogen ---> helium ---> air.

DISCUSSION OF THE EXPERIMENTAL RESULTS

The XPS-analysis verified the presence of the constituents of the coating and also indicated that no impurities were added to the coating during the preparation process.

As previously discussed, friction and wear are the lowest in hydrogen, higher in helium, and highest in air. X-ray diffraction analyses provided a clue for a plausible explanation of this trend.

A comparison of x-ray diffraction peak intensities (peak heights) for coatings tested to 760 °C in the three atmospheres showed that only the chromic oxide level was affected by the test atmosphere. The relative chromium oxide concentration levels were approximated from the intensities of the diffraction peak for the (110) plane of the chromic oxide crystal structure with no correction for absorption or fluorescence. The relative chromic oxide levels were obtained simply by normalizing the peak heights relative to the air atmosphere case.

As would be expected, the relative concentration level of chromic oxide on the wear tracks is highest for specimens run in air and lowest for specimens run in hydrogen. In spite of the high purity of the bottled test gases, chromic oxide is found on the wear tracks of specimens run in helium and in hydrogen due to residual oxygen and possibly water vapor present in the test chamber.

Figure 6 is a graph of the average friction coefficient versus the relative chromic oxide level normalized to the chromic oxide level in air. The data points are limited in number, but the trend indicates that there is a direct relationship between the friction coefficient and the level of chromic oxide present at the sliding interface for this coating system.

Chromic oxide is a hard, refractory compound that is a good anti-wear material when applied as a smooth, adherent coating (ref. 7). In this instance however, the chromic oxide that forms on the plasma sprayed coating apparently acts as an abrasive component in the sliding contact, thereby increasing friction and wear.

To determine other factors influencing the friction and wear behavior of the coating, Energy Dispersive Spectra (EDS) x-ray analyses of the rider wear scars was performed. These analyses indicated that the amount of silver transfer from the parent coating to the rider wear scar is much lower for specimens run in air than in helium or hydrogen. Previous studies indicate that silver transfer has a beneficial effect on the tribological performance of the coating (ref. 5). Thus the lower levels of silver transfer for specimens run in air may be inhibiting its lubricating function in these cases.

It is difficult to prove that the high levels of chromic oxide present on samples run in air, inhibit beneficial silver transfer. It is likely, however, that both high chromic oxide levels on the wear track and reduced silver transfer to the rider wear scar are significant factors contributing to higher friction and wear.

The effect of prefusing the eutectic is evident by comparing the friction and wear data in table IV. Friction coefficients are 15 to 20 percent lower, counterface wear is 20 to 30 percent lower and coating wear remains about the same for coatings prepared by this improved processing method. They are also more uniform and easier to plasma spray. Therefore, prefusing the fluoride eutectic has been adopted as a standard part of the coating procedure.

CONCLUSIONS

1. The friction and wear of a chromium carbide based coating (PS200) is affected by the atmosphere present during sliding. Friction and wear properties are better in inert or reducing atmospheres than in air.

2. The test atmosphere is one factor that controls the formation of chromic oxide which seems to have an adverse effect on the tribological properties of the coating and may be inhibiting beneficial silver transfer.

3. There may be a direct relationship between chromic oxide level present on the wear surfaces and the friction coefficient.

4. The test data indicate that the coating behavior can be improved if the formation of chromic oxide can be reduced and if silver transfer from the parent coating can be optimized.

APPENDIX A

RECOMMENDED GRINDING PROCEDURE

1. Use diamond grinding only.
2. Use water as lubricant--use no oil.
3. Initial grinding depth should be 0.0025 cm.
4. Final cuts should be 0.001 to 0.0015 cm.
   a. Taking too deep a cut, i.e., 0.01 cm, will pluck softer phases (Ag and BaF_2/CaF_2) from surface.
   b. Taking too light a cut, i.e., less than 0.001 cm, will smear the metal-bonded chromium carbide. This will result in an "Orange Peel" type finish.
5. Ground surface should be matte, not glossy, and have a speckled appearance representing the three separate phases.

<u>NOTE</u>: The grinding wheel should be dressed regularly to maintain sharp diamond cutting edges; otherwise the above procedure will not insure the achievement of a satisfactory surface.

APPENDIX B

EXPLANATION OF WEAR FACTORS

The wear factor (k) used in this paper is a coefficient which relates the volume of material worn from a surface to the distance slid and the normal load at the contact. Mathematically, k is defined as:

$$k = V/(S \times W)$$

where

W = the normal load at the sliding contact in kg.
S = the total distance slid in cm.
V = the volume of material worn away in cm^2.

The physical interpretation of the numeric value of the k factor is as follows:

$k = 10^{-8} cm^3/cm\text{-}kg$	high wear.
$k = 10^{-9}$ to $10^{-10} cm^3/cm\text{-}kg$	moderate to low wear.
$k = 10^{-11} cm^3/cm\text{-}kg$	very low wear.

Some authors prefer wear factor units of $mm^3/(N\text{-}m)$. Conversion to these units can be made to a close approximation by multiplying our units by 10^4.

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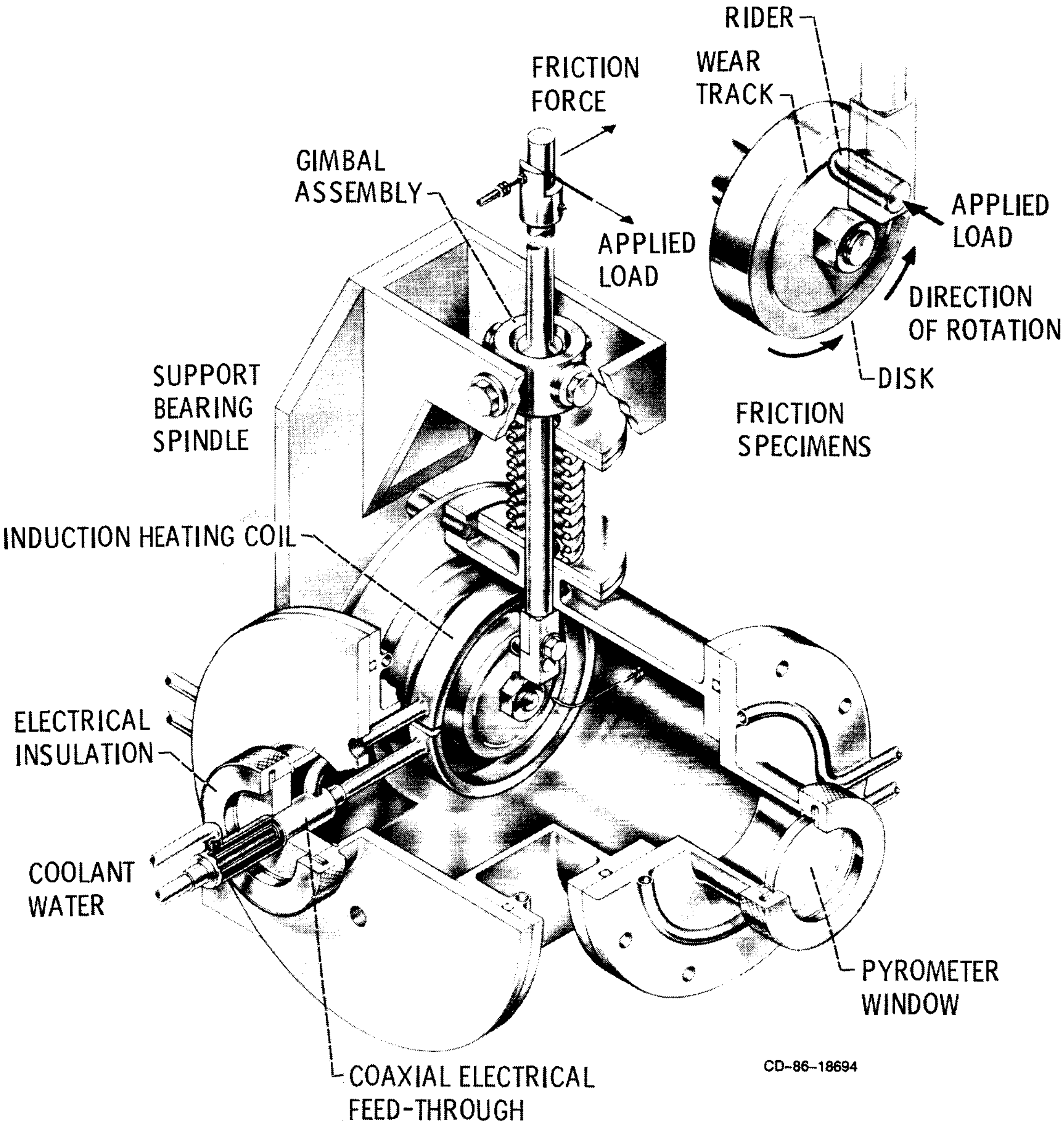

FIGURE 1.- HIGH-TEMPERATURE FRICTION APPARATUS.

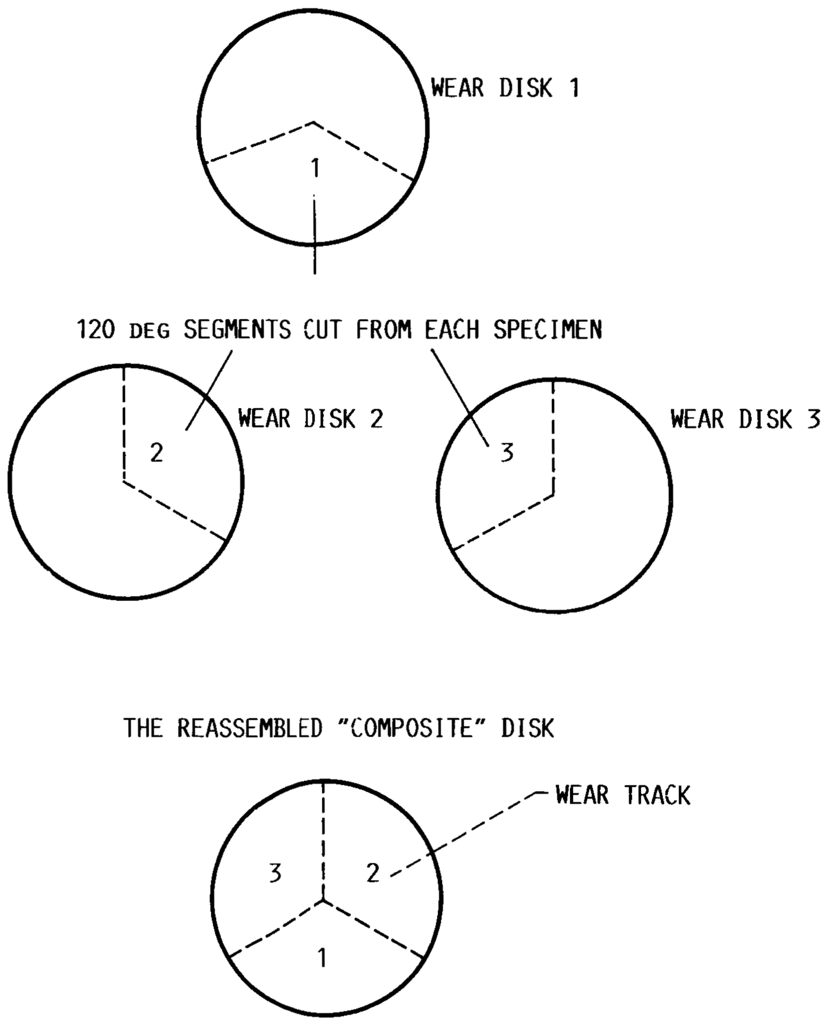

FIGURE 2.- THE XPS X-RAY COMPOSITE SPECIMEN.

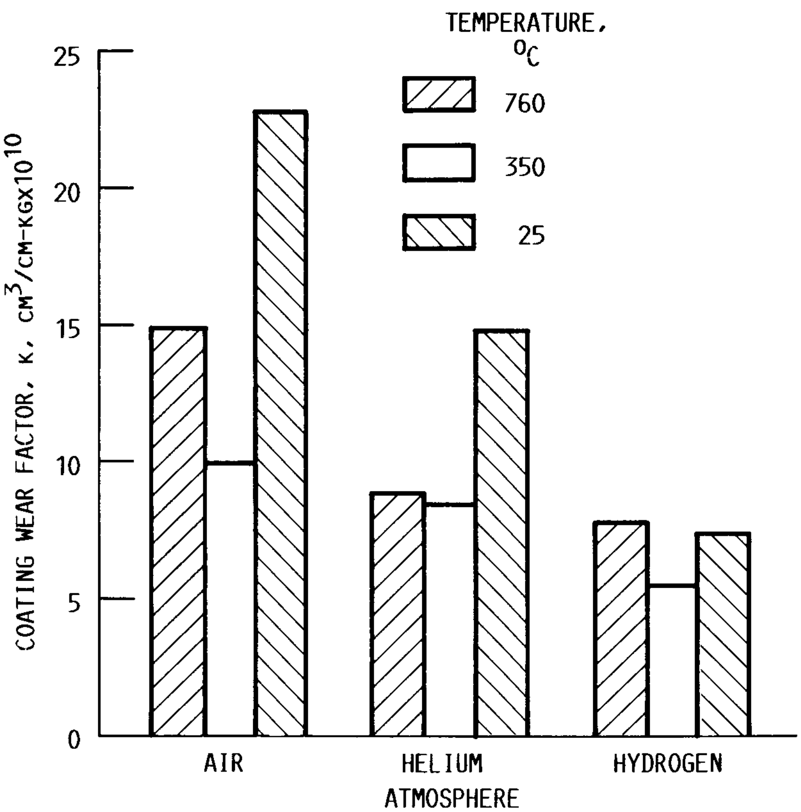

FIGURE 3.- DISK COATING WEAR FACTOR, K, FOR THE PS200 COATING IN VARIOUS ATMOSPHERES. TEST CONDITIONS: 2.7 M/S SLIDING VELOCITY, 38.97 KPA CHAMBER PRESSURE, 0.5 KG NORMAL LOAD.

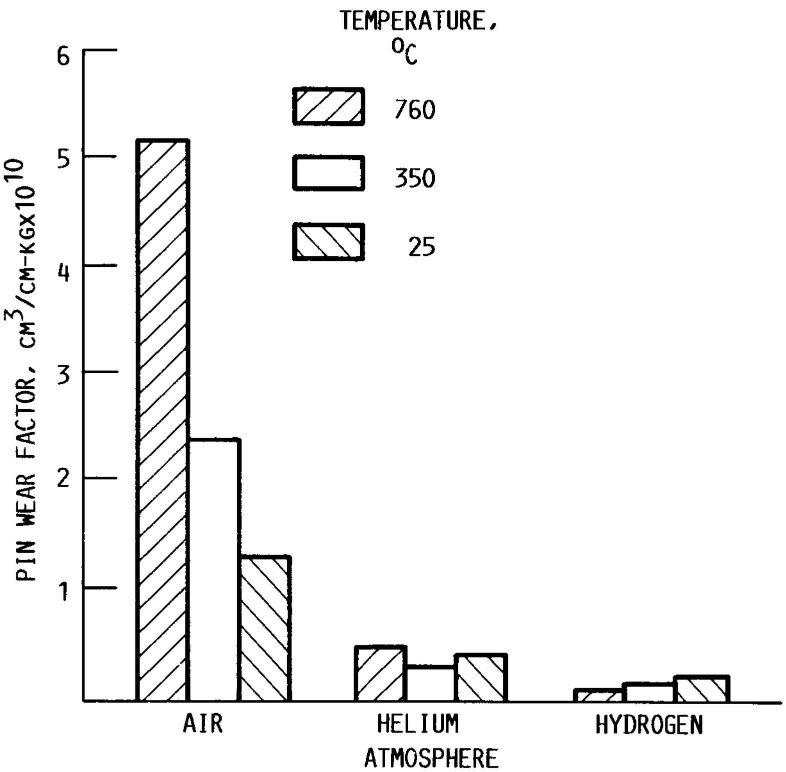

FIGURE 4.- PIN WEAR FACTOR, K, FOR THE HARDENED COBALT ALLOY TESTED AGAINST THE PS200 COATING IN VARIOUS TEST ATMOSPHERES. TEST CONDITIONS: 2.7 M/S SLIDING VELOCITY, 38.97 KPA CHAMBER PRESSURE, 0.5 KG NORMAL LOAD.

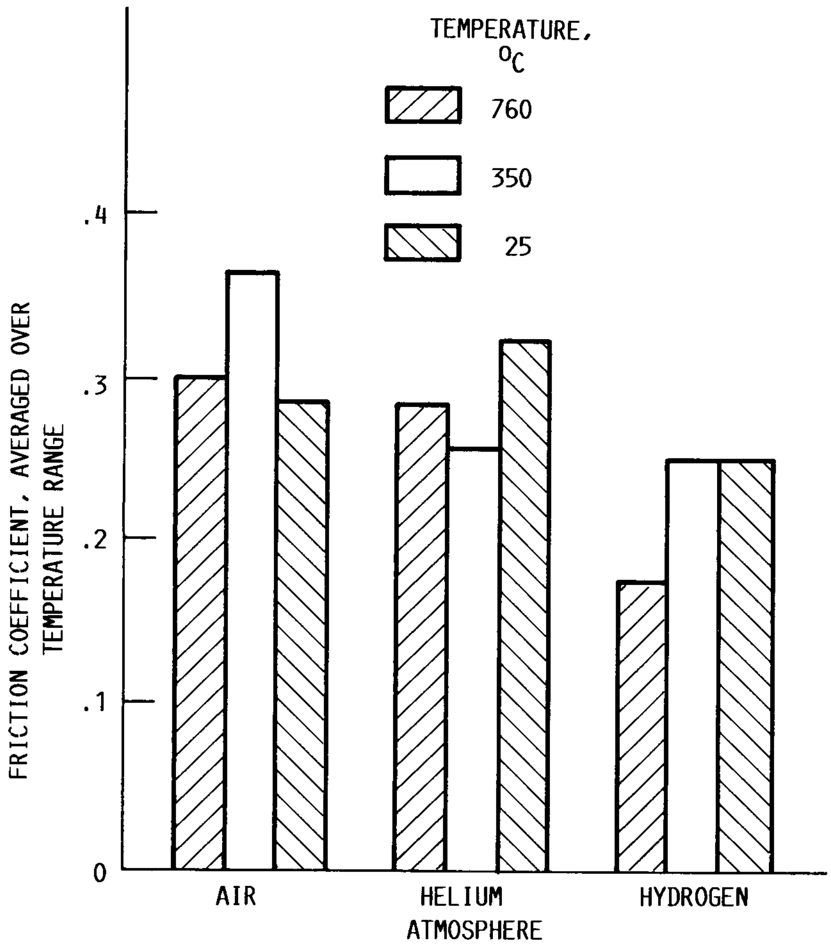

FIGURE 5.- FRICTION COEFFICIENT FOR PS200 SLIDING AGAINST HARDENED COBALT ALLOY IN VARIOUS ATMOSPHERES. TEST CONDITIONS: 2.7 M/S SLIDING VELOCITY, 0.5 KG LOAD, 38.97 KPA CHAMBER PRESSURE.

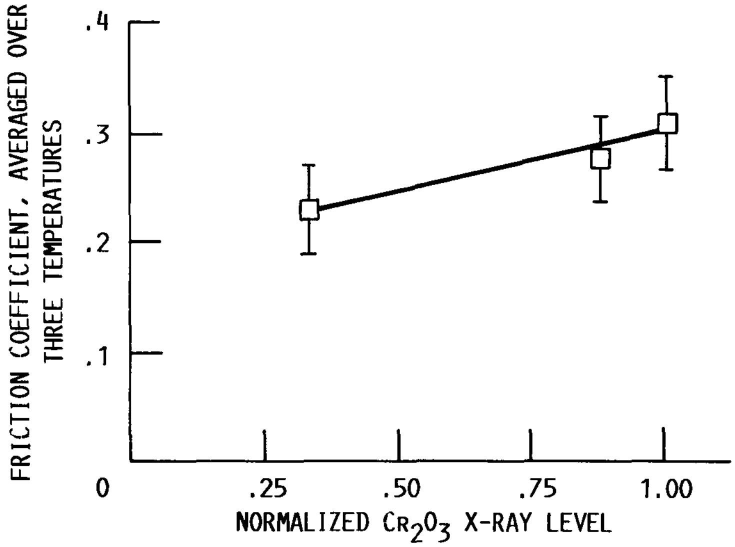

FIGURE 6.- AVERAGED FRICTION COEFFICIENT VERSUS RELATIVE WEAR TRACK CR_2O_3 CONCENTRATION LEVEL NORMALIZED TO THE AIR CONCENTRATION LEVEL. TEST CONDITIONS: 2.7 M/S SLIDING VELOCITY, 0.5 KG LOAD. ERROR BARS REPRESENT ONE STANDARD DEVIATION OF THE DATA POINTS.

TABLE I. - COMPOSITION OF THE THREE MAJOR COATING COMPONENTS

Component	Composition, wt %	Particle
Bonded chromium carbide		
Ni Al Cr_3C_2 Co	28 2 58 12	-200 + 400 Mesh
Silver metal		
Ag	100	-100 + 325
Prefused eutectic		
BaF_2 CaF_2	62 38	-200 + 325
Nonprefused eutectic		
BaF_2 CaF_2	62 38	Reagent Grade Precipitated powder

TABLE II. - TYPICAL PLASMA SPRAY PARAMETERS

Parameter	Material, value
Arc gas 1.4 m^3/hr	Argon
Powder carrier gas	Argon 0.4 m^3/hr
Coating powder flow rate	1 kg/hr
Amperage	450 to 475 A
Voltage	32 V
Gun to specimen distance	approx. 15 cm

TABLE III. - FRICTION AND WEAR SUMMARY OF PS200 SLIDING AGAINST COBALT ALLOY

[Test conditions: 0.5 kg load, 2.7 m/s sliding velocity, 38.97 kPa chamber pressure.]

Test Atmosphere	Temperature, °C	Average friction coefficient, μ	Average rider k factor, $cm^3/kg\ cm$	Average coating k factor, $cm^3/kg\ cm$
H_2	760	0.18±0.05	$0.75x10^{-11}$±0.35	$7.6x10^{-10}$±1.9
H_2	350	.26±.06	$1.25x10^{-11}$±.35	$4.5x10^{-10}$±2.1
H_2	25	.26±.04	$2.0x10^{-11}$±.35	$7.0x10^{-10}$±3.8
Average value	---	.23±.03	$1.33x10^{-11}$±.35	$6.4x10^{-10}$±1.6
Helium	760	.28±.04	$5.62x10^{-11}$±2.4	$8.7x10^{-10}$±.7
Helium	350	.26±.03	$3.44x10^{-11}$±1.9	$8.2x10^{-10}$±.5
Helium	25	.32±.03	$4.17x10^{-11}$±1.2	$1.5x10^{-9}$±.23
Average value	---	.29±.03	$4.4x10^{-11}$±1.2	$1.04x10^{-9}$±.4
Air	760	.30±.03	$5.16x10^{-10}$±1.1	$1.5x10^{-9}$±.2
Air	350	.37±.04	$2.35x10^{-10}$±1.0	$1.0x10^{-9}$±.2
Air	25	.28±.03	$1.31x10^{-10}$±.8	$2.1x10^{-9}$±.2
Average value	---	.32±.05	$2.94x10^{-10}$±2	$1.5x10^{-9}$±.5

TABLE IV. - AVERAGE DATA FOR PS200 COATINGS WITH BOTH PREFUSED AND NONPREFUSED EUTECTIC

[Test conditions: Specimens run in hydrogen atmosphere, 0.5 kg load, 2.7 m/s sliding velocity, slid against hardened cobalt alloy. Values given are averages over temperature range.]

Powder preparation	Nonprefused fluoride eutectic	Prefused fluoride eutectic
Average friction coefficient	0.28	0.23
Average pin wear factor k, $cm^3/(kg\ cm)$	$1.5x10^{-11}$	$1.2x10^{-11}$
Average coating wear factor k, $cm^3/(kg\ cm)$	$6.5x10^{-10}$	$6.3x10^{-10}$

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16. Abstract

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